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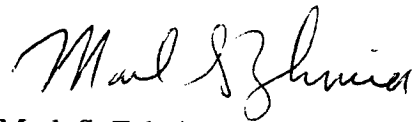
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Enclosed is the final report for contract NAS5-32917 covering the period of July 1995 to June 1998.

Sincerely yours,



Mark S. Zahniser
Principal Research Scientist

cc: Dr. Louis Stief, COTR, Mail Code 690
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**LABORATORY STUDIES OF
CHEMICAL AND PHOTOCHEMICAL
PROCESSES RELEVANT TO
STRATOSPHERIC OZONE**

Prepared by

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ABSTRACT

The purpose of this project is to reduce the uncertainty in rate coefficients for key gas-phase kinetic processes which impact our understanding of stratospheric ozone. The main emphasis of this work is on measuring the rate coefficients for the reactions of $\text{HO}_2 + \text{O}_3$ and $\text{HO}_2 + \text{NO}_2$ in the temperature range (200 to 240 K) relevant to the lower stratosphere. In order to accomplish this, a high pressure turbulent flow tube reactor was built and its flow characteristics were quantified. The instrument was coupled with tunable diode laser spectroscopy for HO_2 detection. Room temperature measurements of the $\text{HO}_2 + \text{NO}_2$ rate coefficients over the pressure range of 50 - 300 Torr agree well with previous measurements. Preliminary measurements of the $\text{HO}_2 + \text{O}_3$ rate coefficients at 300 torr over the temperature range of 208 - 294 K agree with the NASA Evaluation from 294 - 225 K but deviate significantly (50% higher) at ~210 K.

1. INTRODUCTION

Over the past three years we have focused on the development of a turbulent flow tube reactor with infrared absorption detection of HO_2 using a tunable diode laser with an astigmatic multipass cell. Upon completion of the construction of this instrument, the turbulent flow dynamics for the flow reactor were assessed with a Pitot tube flow speed probe. The experimental technique and instrument were then validated by measuring the rate coefficient for the $\text{HO}_2 + \text{NO}_2$ rate coefficient over the pressure range of 50 - 300 Torr. The rate coefficient for the $\text{HO}_2 + \text{O}_3$ reaction was then measured at 300 Torr from 294 - 208 K to attempt to and resolve the long-standing question of the temperature dependence of this reaction at lower stratospheric temperatures. The instrument and kinetic measurements will be described below. In addition to the laboratory work, Dr. Kolb has participated in NASA's panel for Data Evaluation helping to prepare the most recent version of the JPL publication "Chemical Kinetics and Photochemical Data for Use in Stratospheric Modeling" [DeMore et al., 1997].

Comparisons between measurements and modeling using experimentally determined rate coefficients and branching ratios have shown that there are still gaps in our knowledge of stratospheric ozone chemistry. For example, recently reported simultaneous measurements of HO_x , NO_x , ClO , and BrO radicals demonstrate that the uncertainties in ozone removal rates in the 16 to 20 km altitude region are dominated by the uncertainty in laboratory-measured rate coefficients for the catalytic cycles involving these species [Wennberg et al., 1994]. Many of the reactions in question are most amenable to investigation through the use of a flow tube reactor, especially reactions involving HO_2 . The investigation of HO_2 reactions in a flow tube reactor at temperatures (200 - 240 K) characteristic of the lower stratosphere has proven especially difficult due to large HO_2 wall losses at these temperatures and the low pressures (1-10 Torr) of the traditional operation [Howard, 1979, Kaufmann, 1961] of flow tube reactors.

The low pressure operation of a flow tube reactor allows for uniform concentrations across the diameter of the flow tube due to rapid diffusion and makes possible a simple conversion between flow tube length and reaction time using the bulk gas flow speed. At higher pressures, the diffusion across the laminar flows is not sufficient to eliminate the concentration gradients.

Molina and coworkers have demonstrated [Seeley et al., 1993; Seeley, 1994] the use of a flow tube in the turbulent regime as a very useful technique for extending upward the pressure regime of flow tube kinetics. The use of turbulent conditions takes advantage of eddy diffusion and a core velocity profile which is relatively flat to allow for a singular core flow speed to be used to determine reaction time. The core flow speed can be determined empirically by measurements with a Pitot tube probe and has been shown [Seeley et al., 1993; Seeley, 1994] to require only a slight correction (-10%) to the average gas flow speed. The laminar layer at the wall effectively separates the wall from the core flow. This reduces possible problems due to heterogeneous reactions on the flow tube wall and significantly reduces wall loss for species such as HO_2 which are readily lost on the wall at colder temperatures relevant to the lower stratosphere. The simple analysis of turbulent flow tube kinetics makes this technique ideal for investigating reactions important in the lower stratosphere at pressures (50 - 200 Torr) and temperatures (200 - 240 Torr) characteristic of this region of the atmosphere. In an effort to develop a general technique for the investigation of HO_2 reaction kinetics at temperatures and pressures of the lower stratosphere, a turbulent flow tube reactor with tunable diode laser/astigmatic multi-pass cell detection has been constructed and characterized through measurements of the reaction rates for HO_2 with O_3 and with NO_2 .

2. TURBULENT FLOW TUBE REACTOR

The instrument consists of a temperature regulated flow tube reactor with movable injector through which HO_2 radicals are added to the main flow containing the reactant gas. HO_2 radicals are detected by tunable diode laser absorption using an astigmatic Herriott multipass cell [Kebabian, 1994; McManus et al., 1995] into which a fraction of the flow is sampled. A simplified schematic of the instrument is shown in Figure 1.

The reactor is a 2.5 cm ID Pyrex tube which has been coated with a thin layer of halocarbon wax to reduce HO_2 wall loss and can be temperature regulated. N_2 flows of ~ 1300 SCCS ($\text{STP cm}^3 \text{ s}^{-1}$) from a liquid nitrogen gas pack dewar pass through the tube and the majority of this is pumped by a 28 l/s Busch pump. The pressure of the flow tube is maintained at 50 - 300

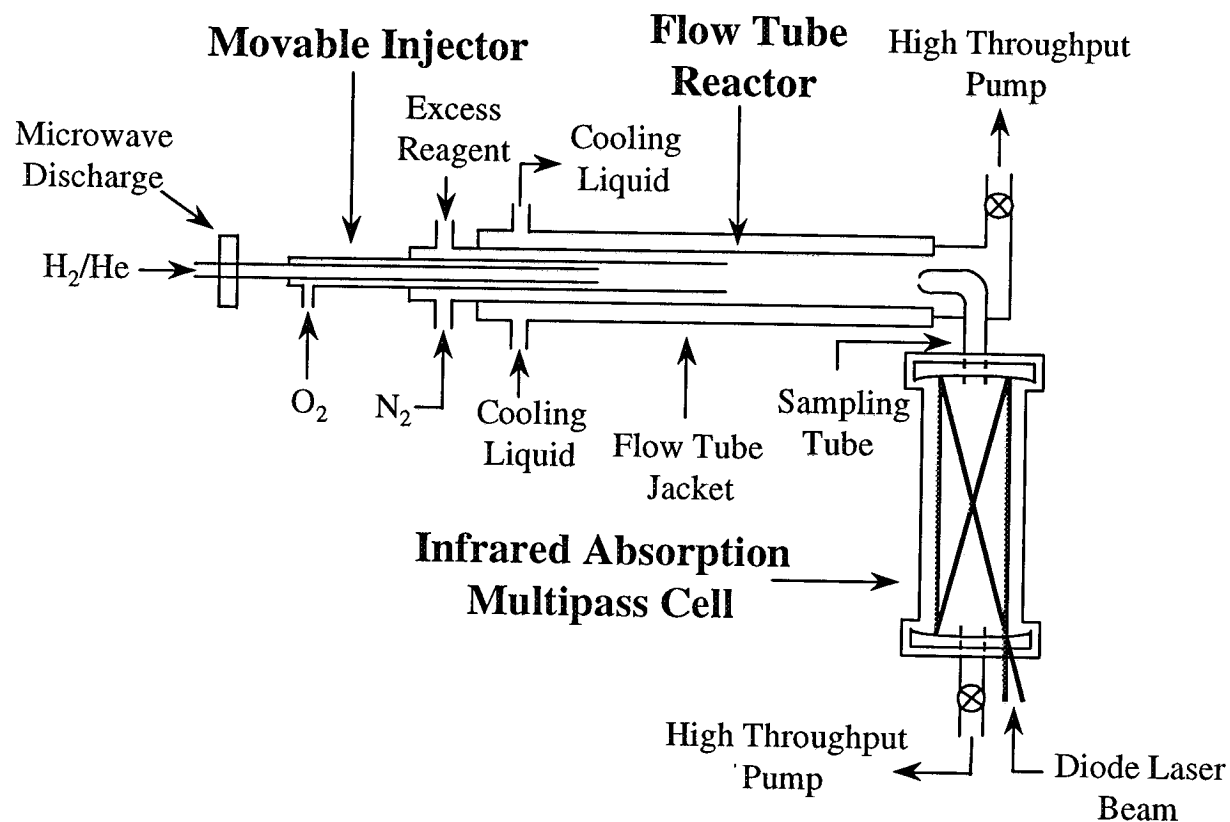


Figure 1. Simple schematic of the turbulent flow tube reactor with infrared tunable diode laser/multipass cell detection.

Torr. The HO₂ radicals ($<1 \times 10^{12}$ molecule cm⁻³) are generated in a movable injector. The HO₂ radicals are generated by reaction of O₂ with H atoms which are produced by passing H₂/He through a 2.5-GHz Beenacker microwave discharge cavity. The reagent gases, NO₂ ($7 \times 10^{12} - 3 \times 10^{14}$ molecule cm⁻³) and O₃ ($0.3 - 2.0 \times 10^{16}$ molecule cm⁻³), enter the flow tube upstream of the injector output. The final 25 cm of the injector is coated with halocarbon wax to reduce the HO₂ injector wall loss.

A sampling tube with a 3 mm diameter orifice draws gas flow from the downstream end of the flow tube into a 55 cm long, 8.5 cm ID multipass cell. The multipass cell is maintained at pressures (5 - 25 Torr). The flow through the cell was 460 SCCS at a flow tube pressure of 200

torr. The experimental conditions were constrained such that the amount of the reaction occurring in the multipass cell relative to that occurring in the flow tube is as small as is practical.

Measurements were made with the diode laser multipassed 366 times for a total path length of 201 m. HO₂ concentrations were determined by measuring the fractional absorption of the HO₂ at a set of nearly coincidental lines at 1411 cm⁻¹. The diode laser frequency is rapidly swept via computer control at 1 kHz with 300 points over the full infrared transition line shape. This scan rate is fast enough to reduce the impact of low frequency noise present on the laser light. The data acquisition system can acquire and process this data stream at 100% duty cycle. The HO₂ production is modulated at 1 Hz by turning the discharge power on and the absolute HO₂ concentration is calculated by fitting the absorption feature to a Voigt profile.

3. FLOW PROFILE CHARACTERIZATION

Fully developed turbulent flow through a tube is characterized by a turbulent core flow with a relatively flat flow speed profile with a thin, slow flowing laminar layer at the flow tube wall. The flow tube lengths used here (10 - 55 cm) are not large enough for the development of turbulent flow so two perforated Teflon “turbulizers” at the tip of the injector are used to even out the flow around the injector and induce turbulence as the bulk of the carrier gas passes by the injector tip and into the reaction distance of the flow tube.

Flow speeds were measured at the downstream end of the flow tube with a Pitot tube. The Pitot tube was moved vertically across the flow tube cross section. The flow speed profiles were measured at different injector positions because the flow turbulence is initiated at the tip of the injector. A typical series of flow profile measurements made at different injector positions is shown in Figure 2.

The flow profiles shown here are relatively flat with the flow speed dropping off slightly near the flow tube wall. The profile appears the most flat when the injector is 20 cm from the Pitot tube. The least flat profile is at the 10 cm position while the 55 cm position profile is closer in shape to that of the 10 cm profile than the 20 cm position.

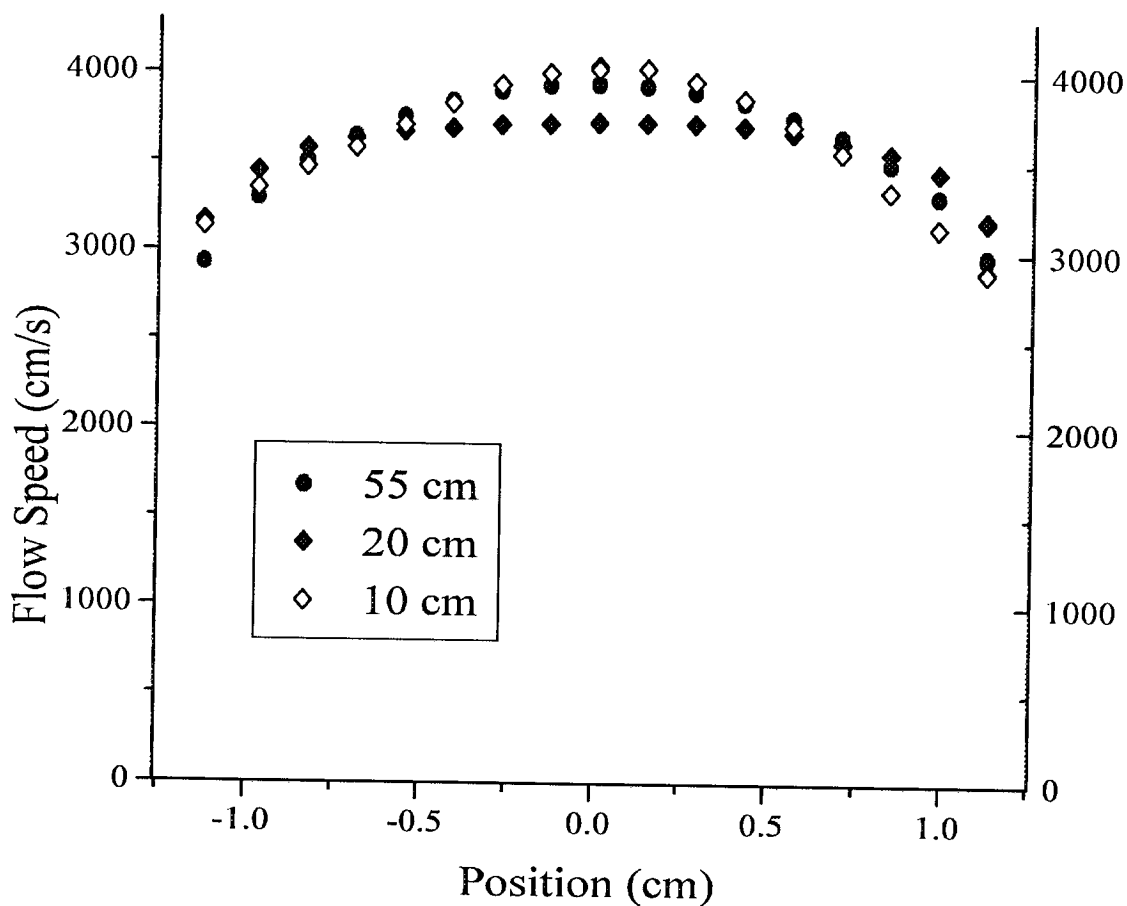


Figure 2. Three flow speed profiles made at distances between the injector and Pitot tube of 10, 20, and 55 cm. The Pitot tube measurements were made as a function of radial position across the width of the flow tube with the 0 cm position corresponding to the center of the flow tube.

The individual flow measurements for a given flow profile were averaged by area weighting to determine a characteristic flow speed (u_{eff}) for the profile. The ratio (u_{eff}/u_{bulk}) of this value and the average bulk flow speed (u_{bulk}) is plotted as a function of Reynolds number (Re) in Figure 3. The data shown is for injector positions from 10 to 55 cm (in 5 cm increments) and at 295, 210, 225, and 230 K. An equation, $u_{eff}/u_{bulk} = 1 + 25.7 \cdot Re^{-0.66}$, was fit to the data at $Re > 3000$ and is used to determine reaction time by calculating the bulk flow speed and Reynolds number

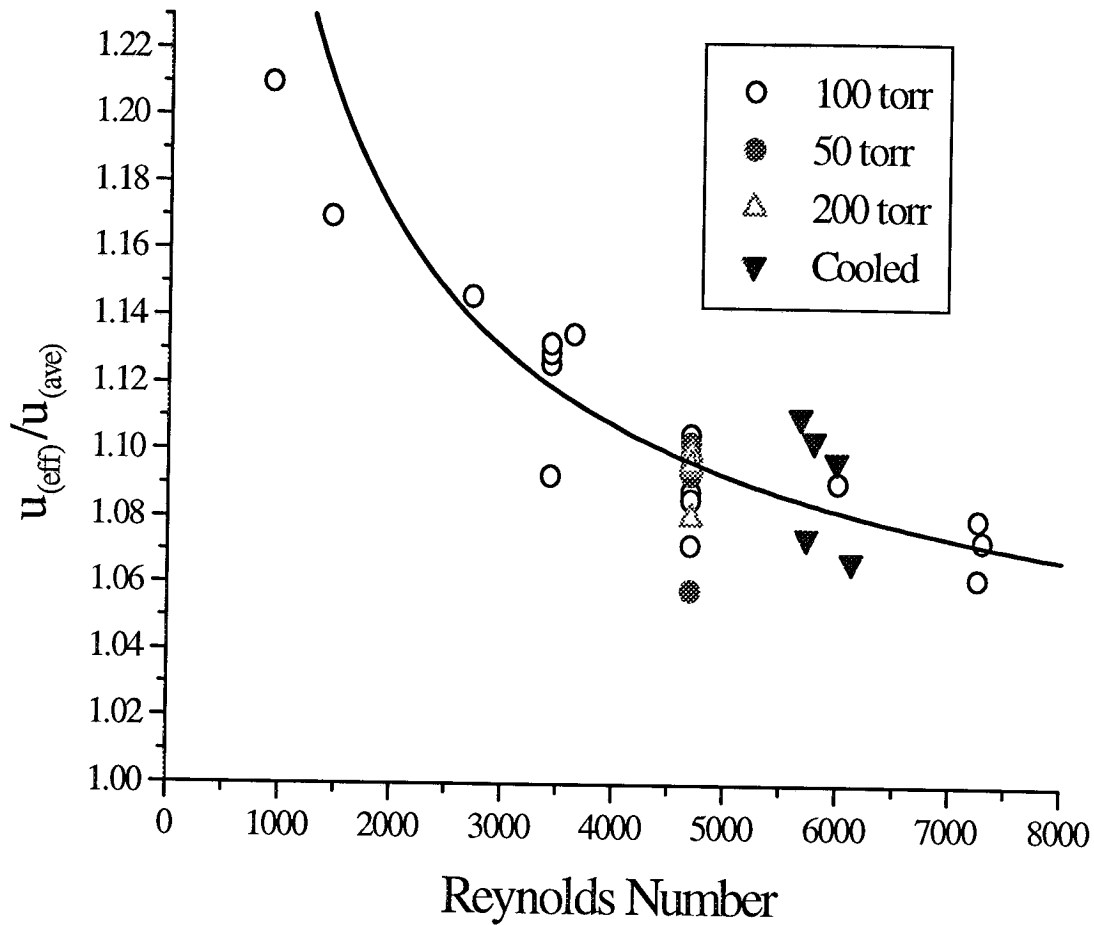


Figure 3. Summary of the ratios of the characteristic flow speed (u_{eff}) for a given profile to the average bulk flow speed (u_{bulk}) plotted as a function of Reynolds Number. The solid triangles represent measurements made at 210, 225, and 230 K. The plotted points are for injector positions from 10 to 55 cm in 5 cm increments. There is no significant trend for temperature, pressure, or injector position.

from the flow tube parameters of a given kinetic measurement. The ratio of the turbulent core flow speed to the bulk flow speed does not vary much with Reynolds numbers greater than 3000. The turbulent core flow speed is on the order of 10% \pm 5% greater than the average bulk speed.

4. HO₂ + NO₂ KINETIC MEASUREMENTS

The rate coefficient for the HO₂ + NO₂ reaction was measured as a function of pressure from 50 - 300 torr at 294 K and 88% N₂ and 12% He. This reaction is important to stratospheric chemistry since it is part of a catalytic cycle which destroys odd-hydrogen radicals. Since this reaction rate has been well studied previously [DeMore et al., 1997], it was chosen as a test case to help validate the turbulent flow reaction technique over this pressure range.

The NO₂ was stored in a blackened 5 L Pyrex bulb with a cold finger. To eliminate any NO or other nitric oxide contamination, 700 Torr of O₂ was added to the NO₂ in the bulb and ~20 hours later the NO₂ was condensed out in the cold finger of the bulb and the remaining gas was pumped off. To eliminate instability of the NO₂ flow due to condensation and evaporation on the needle valve, the cold finger of the bulb was inserted into a water bath maintained at temperatures below the room temperature. The NO₂ flow was measured with a mass flow meter. The NO₂ flow meter was calibrated by diverting the NO₂ flow into a known volume and measuring the pressure change with time. The vapor pressures of NO₂ in the flow tube were < 0.010 Torr which resulted in negligible N₂O₄.

The HO₂ + NO₂ rate coefficients are summarized in Table 1 and shown in Figure 4 plotted as a function of pressure along with previous measurements. As can be seen from Figure 4, the rate coefficients for the HO₂ + NO₂ reaction measured here agree with the previous measurements and the NASA Evaluation. This helps to validate the ability of this instrument and experimental technique to measure HO₂ kinetics over a wide range of pressures.

Table 1. Summary of HO₂ + NO₂ rate coefficient measurements made at 294 K.

Pressure (Torr)	k (cm ³ molecule ⁻¹ s ⁻¹)
50	3.2 x 10 ⁻¹³
101	3.8 x 10 ⁻¹³
151	6.0 x 10 ⁻¹³
201	7.0 x 10 ⁻¹³
300	8.4 x 10 ⁻¹³

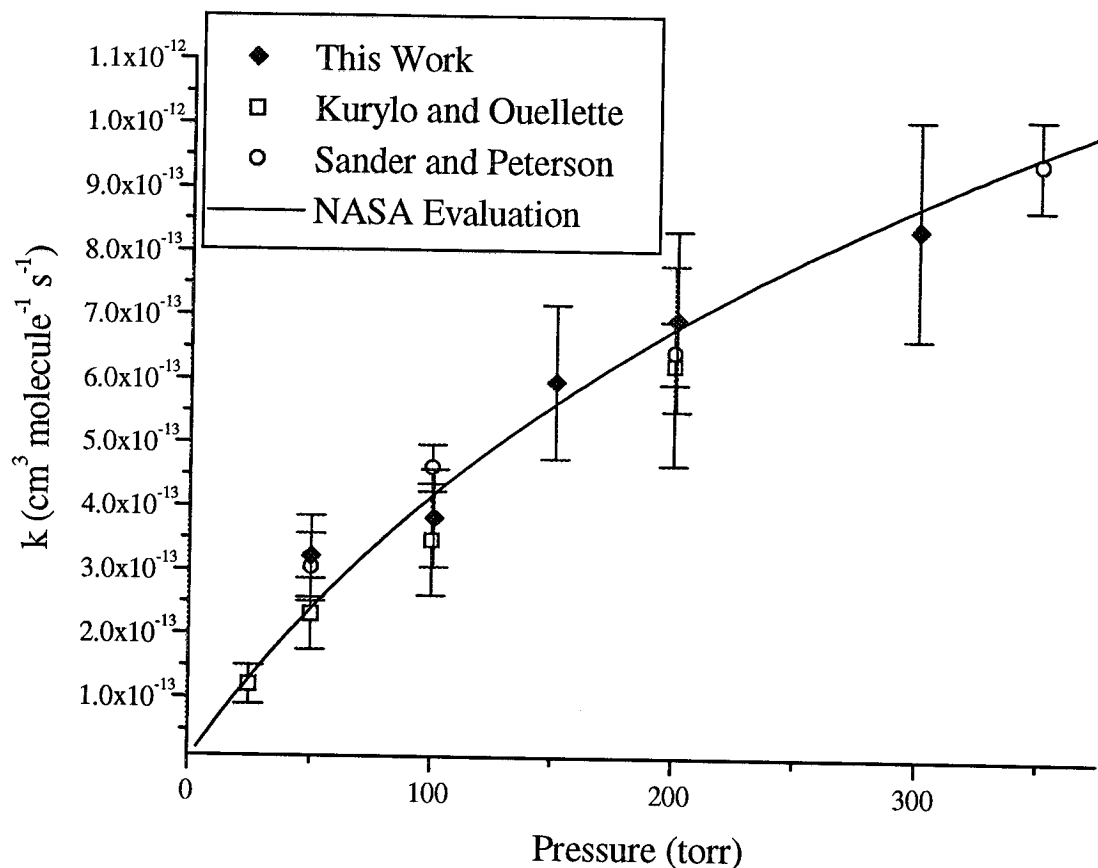


Figure 4. Plot of the $\text{HO}_2 + \text{NO}_2$ rate coefficient as a function of pressure for the measurements made here along with previous measurements. The line shown is the NASA Evaluation recommendation [DeMore et al, 1997].

5. $\text{HO}_2 + \text{O}_3$ KINETIC MEASUREMENTS

The reaction of HO_2 with O_3 is one of the key gas-phase reactions of the lower stratosphere. It is the rate limiting step in an important catalytic ozone destruction cycle which includes the reaction of OH and O_3 to reform HO_2 . This process is particularly significant in the lower stratosphere because it destroys ozone catalytically without requiring atomic oxygen. Recent measurements in the mid-latitude lower stratosphere verify the importance of this process, finding that 30-50% of the ozone removal in this region proceeds through the HO_x pathway

[Wennberg et al., 1994]. An equally important aspect of these reactions is their role in the determination of the partitioning of the HO_x family between OH and HO₂. This partitioning is largely controlled by the HO₂ + O₃, OH + O₃, and HO₂ + NO reactions. Thus, it is of critical importance to measure the rate of the HO₂ + O₃ reaction at stratospheric temperatures and pressures and to reduce the rather large uncertainties [DeMore et al., 1997] associated with the rate of this reaction at temperatures near 200 K.

The use of the turbulent flow tube reactor has allowed for the measurement of the rate coefficient for the HO₂ + O₃ reaction at a pressure (300 torr) more characteristic of the lower stratosphere than the low pressure (<10 torr) measurements made previously. It has also allowed for measurements at lower temperatures (227 - 208 K) than previously possible. This is due to the lower HO₂ wall losses (10 - 15 s⁻¹) at low temperatures (230 - 205 K).

The O₃ used in the kinetic measurements was generated by flowing O₂ through an ozone generator. The resulting O₃/O₂ mixture passed through a ~3 L glass bulb filled with silica gel submerged in ethanol cooled to ~-100°C and the O₃ was trapped in the silica gel. During the kinetic measurements, the O₃ was removed from the silica gel trap by eluting with a flow of N₂. The O₃/N₂ mixture from the trap enters an absorption cell where the partial pressure of the O₃ is measured by absorption at 253.7 nm in a 1.00 cm long cell ($\sigma_{253.7\text{nm}} = 1.15 \times 10^{-17} \text{ cm}^2$) [DeMore et al., 1997]. The O₃ enters the flow tube upstream of the movable injector and the O₃ concentrations in the flow tube were 0.3 - 2.0 × 10¹⁶ molecule cm⁻³. C₂F₃Cl (5 × 10¹⁵ molecule cm⁻³) was added to the flow tube to scavenge the OH radicals produced in the HO₂ + O₃ reaction to prevent OH from reacting with O₃ to reform HO₂.

The rate coefficient for the HO₂ + O₃ reaction was measured at flow tube temperatures of 208 - 294 K. The results of these measurements are plotted together with previous measurements of this reaction in Figure 5. At temperatures below 250 K there appears to be curvature in the Arrhenius plot and an increase in the observed rate with decreasing temperature.

A comparison of the preliminary rate coefficient measurements [Villalta et al., in preparation] made here with the NASA Evaluation recommendation indicates a difference of 50% at the temperatures found in the lower stratosphere. An investigation into the validity of the measurements with regard to the use of the OH scavenger at the colder temperatures is currently

being performed. Experiments where the OH scavenger concentration was varied did not indicate any dependence of the rate coefficient measurements on OH scavenger concentration except at low scavenger concentrations where there was insufficient scavenger to consume the OH produced. A kinetic measurement of the $\text{HO}_2 + \text{O}_3$ reaction at ~ 210 K utilizing isotopically

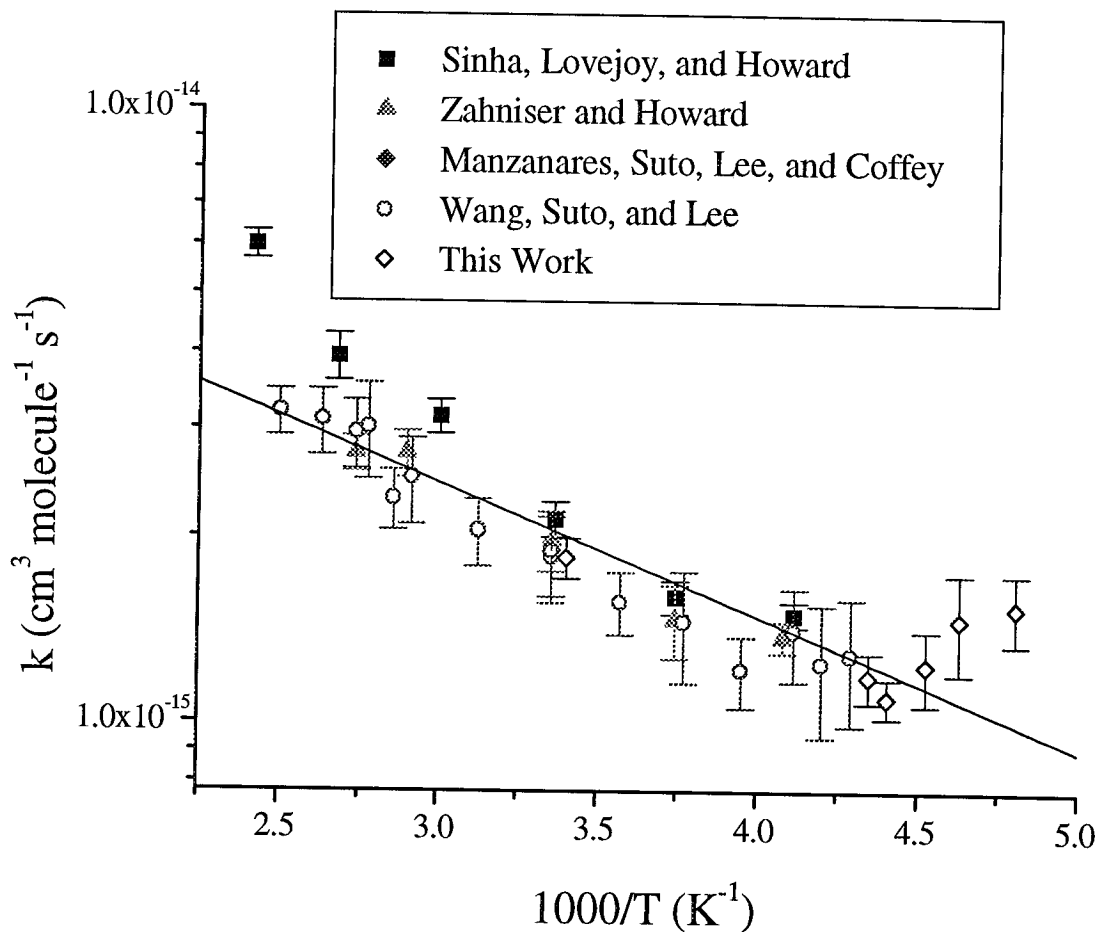


Figure 5. Arrhenius plot of our $\text{HO}_2 + \text{O}_3$ measurements along with the previous measurements. The line is the current NASA Evaluation recommendation.

labeled $O_2(^{18}O_2)$, instead of the OH scavenger, is currently being attempted. If this measurement agrees with the data acquired using the OH scavenger, a reassessment of the $HO_2 + O_3$ rate coefficient at low temperatures appears necessary with potentially significant ramifications for the modeling of ozone levels in the stratosphere.

6. NASA DATA EVALUATION PANEL ACTIVITY

During our current contract C. E. Kolb has served on NASA's Panel for Data Evaluation as the panel's lead member responsible for heterogeneous processes. Due to his efforts on the Heterogeneous Chemistry section of the biennial review, Chemical Kinetics and Photochemical Data for Use in Stratospheric Modeling has been completely revised and significantly expanded in the twelfth edition. The Heterogeneous Chemistry section includes three data tables covering Mass Accommodation Coefficients, Gas/Surface Reaction Probabilities, and Henry's Law Constants for Gas-Liquid Solubilities, as well as text and extensive notes presenting background and guidance for using the tables.

7. ARCHIVAL PUBLICATIONS PREPARED UNDER CONTRACT NO. NAS5-32917

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8. PRESENTATIONS GIVEN UNDER CONTRACT NO. NAS5-32917

Villalta, P.W., M.S. Zahniser, D.D. Nelson, J.T. Jayne and C.E. Kolb, "Gas Phase Kinetic Measurements of the $\text{HO}_2 + \text{NO}$, $\text{HO}_2 + \text{NO}_2$, and $\text{HO}_2 + \text{O}_3$ Reactions at Stratospheric Temperatures and Pressures using a New Turbulent Flow Tube Reactor with Tunable Diode Laser/Astigmatic Multi-Pass Cell Detection", Fourth International Conference on Chemical Kinetics, Gaithersburg, MD, July 14-18, 1997.

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